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DB=USPT,JPAB,EPAB,DWPI; PLUR=YES; OP=ADJ

L19	(cyclohexene) with ((end or terminal)(group))	18	L19
L18	(vinylcyclohexene or vinyl cyclohexene)with ((end or terminal)(group))	0	L18
L17	L16 and (vinylcyclohexene or vinyl cyclohexene)	19	L17
L16	L15 and ((terminal or end)(group))	359	L16
L15	(living or atom transfer or controlled)polymerization	1827	L15

DB=USPT; PLUR=YES; OP=ADJ

L14	L13 and ((terminal or end)(group))	1	L14
L13	5763548.pn.	1	L13

DB=EPAB,DWPI; PLUR=YES; OP=ADJ

L12	802934	3	L12
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DB=USPT; PLUR=YES; OP=ADJ

L11	L10 and (living or controlled or atom transfer)	1	L11
L10	(4808664 or 5523359).pn.	2	L10
L9	l1 and cycl\$2 olefin	1	L9
L8	L5 and terminal group	7	L8
L7	L5 and teminal group	0	L7
L6	L5 and teminal functional group	0	L6
L5	L1 and (linving or controlled)	14	L5
L4	L1 and cyclohexene	0	L4
L3	L1 and (vinylcyclohexene or vinyl cyclohexene)	0	L3
L2	L1 and (vinylcyclohehene or vinyl cyclohexene)	0	L2
L1	(5789487 or 5945491 or 6111022 or 5852129 or 6274688 or 6194516 or 6124411 or 6162882 or 6482900 or 6458903 or 6479584 or 642492 or 6423787 or 5968014 or 6441101).pn.	14	L1

END OF SEARCH HISTORY

WEST**Collections**
Definition, Editing, Browsing**Name:** Undefined**Contents:**

5916989
6310163
6218493
JP402145615 A

Comment:**Database:**

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L17: Entry 2 of 19

File: USPT

Oct 30, 2001

DOCUMENT-IDENTIFIER: US 6310163 B1

TITLE: .alpha.-olefins and olefin polymers and processes therefor

Abstract Text (1):

Disclosed herein are processes for polymerizing ethylene, acyclic olefins, and/or selected cyclic olefins, and optionally selected olefinic esters or carboxylic acids, and other monomers. The polymerizations are catalyzed by selected transition metal compounds, and sometimes other co-catalysts. Since some of the polymerizations exhibit some characteristics of living polymerizations, block copolymers can be readily made. Many of the polymers produced are often novel, particularly in regard to their microstructure which gives some of them unusual properties. Numerous novel catalysts are disclosed, as well as some novel processes for making them. The polymers made are useful as elastomers, molding resins, in adhesives, etc. Also described herein is the synthesis of linear .alpha.-olefins by the oligomerization of ethylene using as a catalyst system a combination a nickel compound having a selected .alpha.-diimine ligand and a selected Lewis or Bronsted acid, or by contacting selected .alpha.-diimine nickel complexes with ethylene.

Brief Summary Text (13):

This invention also concerns a polyethylene containing the structure (XXVII) in an amount greater than can be accounted for by end groups, and preferably at least 0.5 or more of such branches per 1000 methylene groups than can be accounted for by end groups. ##STR2##

Brief Summary Text (14):

This invention also concerns a polypropylene containing one or both of the structures (XXVIII) and (XXIX) and in the case of (XXIX) in amounts greater than can be accounted for by end groups. Preferably at least 0.5 more of (XXIX) branches per 1000 methylene groups than can be accounted for by end groups, and/or at least 0.5 more of (XXVIII) per 1,000 methylene groups are present in the polypropylene. ##STR3##

Brief Summary Text (98):

This invention includes a process for the production of polyolefins, comprising contacting, at a temperature of about -100.degree. C. to about +200.degree. C., one or more monomers selected from the group consisting of ethylene, an olefin of the formula $R_{17}CH_2CH_2$ or $R_{17}CH_2CH(R_{17})$, 4-vinylcyclohexene, cyclobutene, cyclopentene, substituted norbornene, and norbornene; with a compound of the formula ##STR9##

Brief Summary Text (143):

and one or more monomers selected from the group consisting of ethylene, an olefin of the formula $R_{17}CH_2CH_2$ or $R_{17}CH_2CH(R_{17})$, 4-vinylcyclohexene, cyclobutene, cyclopentene, substituted norbornene, or norbornene;

Brief Summary Text (598):

The first compound and (I) are contacted, usually in the liquid phase, and in the presence of the olefin, and/or 4-vinylcyclohexene, cyclopentene, cyclobutene, substituted norbornene, or norbornene. The liquid phase may include a compound added just as a solvent and/or may include the monomer(s) itself. The molar ratio of first

compound:nickel or palladium complex is about 5 to about 1000, preferably about 10 to about 100. The temperature at which the polymerization is carried out is about -100.degree. C. to about +200.degree. C., preferably about -20.degree. C. to about +80.degree. C. The pressure at which the polymerization is carried out is not critical, atmospheric pressure to about 275 MPa, or more, being a suitable range. The pressure may affect the microstructure of the polyolefin produced (see below).

Brief Summary Text (657):

The group T.sup.3 in (VI) was originally the group T.sup.1 in (II) or (III), or the group which included R.sup.8 in (IV). It in essence will normally be one of the end groups of the eventual polymer product. The olefinic group which is coordinated to M, R.sup.11 CH.dbd.CHR.sup.11 is normally one of the monomers, olefin, cyclic olefin, or, if Pd(II) is M, an olefinic ester or carboxylic acid. If more than one of these monomers is present in the reaction, it may be any one of them. It is preferred that T.sup.3 is alkyl and especially preferred that it is methyl, and it is also preferred that R.sup.11 is hydrogen or n-alkyl. It is also preferred that M is Pd(II).

Brief Summary Text (685):

These polymers, and others herein, can have unique structures in terms of the branching in the polymer. Branching may be determined by NMR spectroscopy (see the Examples for details), and this analysis can determine the total number of branches, and to some extent the length of the branches. Herein the amount of branching is expressed as the number of branches per 1000 of the total methylene (--CH.sub.2 --) groups in the polymer, with one exception. Methylene groups that are in an ester grouping, i.e. --CO.sub.2 R, are not counted as part of the 1000 methylenes. These methylene groups include those in the main chain and in the branches. These polymers, which are E homopolymers, have a branch content of about 80 to about 150 branches per 1000 methylene groups, preferably about 100 to about 130 branches per 1000 methylene groups. These branches do not include polymer end groups. In addition the distribution of the sizes (lengths) of the branches is unique. Of the above total branches, for every 100 that are methyl, about 30 to about 90 are ethyl, about 4 to about 20 are propyl, about 15 to about 50 butyl, about 3 to about 15 are amyl, and about 30 to about 140 are hexyl or longer, and it is preferred that for every 100 that are methyl, about 50 to about 75 are ethyl, about 5 to about 15 are propyl, about 24 to about 40 are butyl, about 5 to 10 are amyl, and about 65 to about 120 are hexyl or larger. These E homopolymers are often amorphous, although in some there may be a small amount of crystallinity.

Brief Summary Text (702):

Some of the polymers produced herein are novel because of unusual structural features. Normally, in polymers of alpha-olefins of the formula CH.sub.2.dbd.CH(CH.sub.2).sub.a H wherein a is an integer of 2 or more made by coordination polymerization, the most abundant, and often the only, branches present in such polymers have the structure --(CH.sub.2).sub.a H. Some of the polymers produced herein are novel because methyl branches comprise about 25% to about 75% of the total branches in the polymer. Such polymers are described in Examples 139, 162, 173 and 243-245. Some of the polymers produced herein are novel because in addition to having a high percentage (25-75%) of methyl branches (of the total branches present), they also contain linear branches of the structure --(CH.sub.2).sub.n H wherein n is an integer of six or greater. Such polymers are described in Examples 139, 173 and 243-245. Some of the polymers produced herein are novel because in addition to having a high percentage (25-75%) of methyl branches (of the total branches present), they also contain the structure (XXVI), preferably in amounts greater than can be accounted for by end groups, and more preferably greater than 0.5 (XXVI) groups per thousand methyl groups in the polymer greater than can be accounted for by end groups. ##STR72##

Brief Summary Text (725):

Since it is believed that many of the polymerization processes herein have characteristics that often resemble those of living polymerizations, making block polymers may be relatively easy. One method is to simply allow monomer(s) that are being polymerized to be depleted to a low level, and then adding different monomer(s) or the same combination of monomers in different ratios. This process may be repeated to obtain polymers with many blocks.

Brief Summary Text (797):

Copolymers of linear olefins with 4-vinylcyclohexene and other dienes may generally be used for all of the applications for which the linear olefins polymers (listed above) may be used. In addition they may be sulfur cured, so they generally can be used for any use for which EPDM polymers are used, assuming the olefin/4-vinylcyclohexene polymer is elastometric.

Detailed Description Text (367):

Polymerization of ethylene was carried out according to example 112, using the complex [(2,6-i-PrPh).sub.2 DABMe.sub.2]PdMe(OSO.sub.2 CF.sub.3) (0.068 g, 0.10 mmol) as the initiator and CHCl.sub.3 as the solvent. The reaction gave 5.98 g of low viscosity fluid (2130 equivalents of ethylene per Pd). .sup.1 H NMR (CDCl.sub.3) 0.8-1.0 (m, CH.sub.3); 1.0-1.5 (m, CH and CH.sub.2); 1.5-1.7 (m, CH.sub.3 CH.dbd.CH--); 1.9-2.1 (broad, --CH.sub.2 CH.dbd.CHCH.sub.2 --); 5.3-5.5 (m, --CH.dbd.CH--). Integration of the olefin end groups assuming one olefin per chain gives M.sub.n =630 (DP=24). A linear polymer with this molecular weight and methyl groups at both ends should have 46 methyl carbons per 1000 methylene carbons. The value measured by integration is 161, thus this polymer is highly branched.

Detailed Description Text (369):

Polymerization of ethylene was carried out according to example 112, using the complex {[(2,6-i-PrPh).sub.2 DABH.sub.2]PdCH.sub.2 CH.sub.2 CH.sub.2 C(O)OCH.sub.3 }SbF.sub.6.sup.- (0.082 g, 0.10 mmol) as the initiator and CHCl.sub.3 as the solvent. The reaction gave 4.47 g of low viscosity fluid (1600 equivalents of ethylene per Pd). .sup.1 H NMR (CDCl.sub.3) is similar to example 120. Integration of the olefin end groups assuming one olefin per chain gives M.sub.n =880 (DP32 31). A linear polymer with this molecular weight and methyl groups at both ends should have 34 methyl carbons per 1000 methylene carbons. The value measured by integration is 156, thus this polymer is highly branched.

Detailed Description Text (371):

Polymerization of ethylene was carried out according to example 112, using the complex {[(2,6-i-PrPh).sub.2 DABMe.sub.2]PdCH.sub.2 CH.sub.2 CH.sub.2 C(O)OCH.sub.3 }BCl(C.sub.6 F.sub.5).sub.3.sup.- (0.116 g, 0.10 mmol) as the initiator and CHCl.sub.3 as the solvent. The reaction gave 0.278 g of low viscosity fluid, after correcting for the catalyst residue this is 0.160 g (57 equivalents of ethylene per Pd). M.sub.n estimated by integration of olefin end groups is 300.

Detailed Description Text (373):

The complex [(2,6-i-PrPh).sub.2 DABMe.sub.2]PdMeCl (0.056 g, 0.10 mmol) was loaded into a Schlenk flask in the drybox followed by 40 ml of dry toluene. A solution of ethyl aluminum dichloride (1.37 ml of 0.08 M solution in o-dichlorobenzene) was added while stirring. Polymerization of ethylene was carried out using this solution according to example 112. The reaction gave 0.255 g of low viscosity fluid, after correcting for the catalyst residue this is 0.200 g (71 equivalents of ethylene per Pd). M.sub.n estimated by integration of olefin end groups is 1300.

Detailed Description Text (400):

GPC was done in THF using PMMA standards and an RI detector except for example 133 which was done in trichlorobenzene at 135.degree. C. vs. polystyrene reference with results calculated as linear polyethylene using universal calibration theory. When polymer end groups could be detected by .sup.1 H NMR (5.4 ppm, multiplet, --CH.dbd.CH--, internal double bond), M.sub.n was calculated assuming two olefinic protons per chain.

Detailed Description Text (488):

The solid complex {[(2,6-i-PrPh).sub.2 DABH.sub.2]PdMe(Et.sub.2 O)}SbF.sub.6.sup.- (0.080 g, 0.10 mmol) was added as a solid to a stirring solution of cyclopentene (1.35 g, 20 mmol) in 20 ml of dichlorobenzene in the drybox. After stirring 20 h, the slightly viscous solution was worked up by removing the solvent in vacuo to give 1.05 g sticky solid (156 equivalents of cyclopentene per Pd). .sup.1 H NMR (CDCl.sub.3): complex spectrum from 0.6-2.6 ppm with maxima at 0.75, 1.05, 1.20, 1.55, 1.65, 1.85, 2.10, 2.25, and 2.50. There is also a multiplet for internal olefin at 5.25-5.35. This is consistent with a trisubstituted cyclopentenyl end

group with a single proton (W. M. Kelly et al., *Macromolecules* 1994, 27, 4477-4485.) Integration assuming one olefinic proton per polymer chain gives DP=8.0 and Mn=540. IR (Thin film between NaCl plates, cm.sup.-1): 3048 (vw, olefinic end group, CH stretch), 1646(vw, olefinic end group, R.sub.2 C.dbd.CHR trisubstituted double bond stretch), 1464(vs), 1447(vs), 1364(m), 1332(m), 1257(w), 1035(w), 946(m), 895(), 882(w), 803 (m, cyclopentenyl end group, R.sub.2 C.dbd.CHR trisubstituted double bond, CH bend), 721 (vw, cyclopentenyl end group, RHC.dbd.CHR disubstituted double bond, CH bend). GPC (trichlorobenzene, 135.degree. C., polystyrene reference, results calculated as linear polyethylene using universal calibration theory): M.sub.n =138 M.sub.w =246 M.sub.w /M.sub.n =1.75.

Detailed Description Text (642):

Toluene (30 mL), 4-vinylcyclohexene (15 mL), and 20 mg of [(2,6-i-PrPh).sub.2 DABH.sub.2]NiBr.sub.2 (0.03 mmol) were combined in a Schlenk flask under an atmosphere of ethylene. A 10% MAO solution (3 mL) in toluene was added. The resulting purple solution was stirred for 16 h. After only a few hours, polymer began to precipitate and adhere to the walls of the flask. The polymerization was quenched and the polymer precipitated from acetone. The polymer was dried in vacuo overnight resulting in 100 mg of a white solid. Characterization by proton NMR suggests in corporation of 4-vinylcyclohexene as a comonomer. .sup.1 H NMR (CDCl.sub.3) .delta. 5.64 (m, vinyl, cyclohexene), 2.0-0.9 (overlapping m, including cyclohexyl methylene, methylene (PE), methine), 0.78 (methyl, PE). There are also some minor signals in the base line that suggests incorporation of the internal olefin (cyclohexene) and free .alpha.-olefin (4-vinyl).

Detailed Description Text (729):

A 26-mg (0.033-mmol) sample of [(2,6-i-PrPh).sub.2 DAB(4-F-Ph).sub.2]NiBr.sub.2 was magnetically stirred under nitrogen in a 50-mL Schlenk flask with 25 mL of dry toluene. Then 0.6 mL of polymethylalumoxane was injected, turning the orange-brown solution to a deep green-black solution. The mixture was pressurized immediately with ethylene to 152 kPa (absolute) and stirred at RT for 17 h. The reaction soon became warm to the touch; this heat evolution persisted for over an hour and the liquid volume in the Schlenk flask was observed to be slowly increasing. After 17 h, the reaction was still dark green-brown, but thicker and significantly (20%) increased in volume. The ethylene was vented; the offgas contained about 3% butenes (1-butene, 1.9%; t-2-butene, 0.6%; c-2-butene, 0.9%) by GC (30-m Quadrex GSQ Megabore column; 50-250.degree. C. at 10.degree./min). The toluene solution was stirred with 6N HCl/methanol and was separated; the toluene was rotovapped and held under high vacuum to yield 9.53 g of low-melting polyethylene wax. There seemed to be significant low-boiling species present, probably low-mw ethylene oligomers, which continued to boil off under high vacuum. .sup.1 H NMR (CDCl.sub.3 ; 60.degree. C.) of the product showed a CH.sub.2 :CH.sub.3 ratio of 206:17, which is 57 CH.sub.3 's per 1000 CH.sub.2 's. There were vinyl peaks at 5-5.8 ppm; if the end groups are considered to be vinyls rather than internal olefins, the degree of polymerization was about 34.

Detailed Description Text (790):

Cyclopentene was polymerized by [(2-PhPh).sub.2 DABMe.sub.2]NiBr.sub.2 and MMAO according to Example 305 to give 1.24 g polymer (728 turnovers/Ni). The polymer was pressed at 292.degree. C. to give a transparent, light gray, brittle film. DSC (25 to 320.degree. C., 10.degree. C./min, second heat): Tm(onset)=160.degree. C., Tm(end)=285.degree. C., Heat of fusion=33 J/g. .sup.1 H NMR (400 MHz, 120.degree. C., d.sub.4 -o-dichlorobenzene) is very similar to the spectrum of Example 306. Several peaks attributed to cyclopentenyl end groups were observed in the range 5.2-5.7 ppm. Integration of these peaks was used to calculate M.sub.n =2130. IR (pressed film, cm.sup.-1): 3050 (vw, olefinic end group, CH stretch), 1615(vw, olefinic end group, cis-CH.dbd.CH-- double bond stretch), 1463(vs), 1445(vs), 1362(s), 1332(s), 1306(s), 1253(m), 1128(w), 1041(w), 935(m), 895(w), 882(w), 792(w), 721(w, olefinic end group, cis-CH.dbd.CH--, CH bend). GPC (Dissolved in 1,2,4-trichlorobenzene at 150.degree. C., run at 100.degree. C. in tetrachloroethylene, polystyrene calibration): Peak MW=13,900; M.sub.n =10,300; M.sub.w =17,600; M.sub.w /M.sub.n =1.70.

Detailed Description Text (1010):

Palladium Polymerization Procedure: (0.06 mmol scale). Polyethylene (0.06 g) was

isolated as an oil. ¹H NMR spectrum (C₆D₆) shows branched polyethylene along with some internal olefinic end groups.

Detailed Description Text (1017):

Nickel Polymerization Procedure: (0.02 mmol scale) solid white polyethylene (5.9 g) was isolated. ¹H NMR spectrum (C₆D₆) shows a significant amount of branched polymer along with internal olefinic end groups.

Detailed Description Text (1018):

Palladium Polymerization Procedure: (0.06 mmol scale) Polyethylene (0.38 g) was isolated as an oil. ¹H NMR spectrum (C₆D₆) shows a significant amount of branched polymer along with internal olefinic end groups.

Detailed Description Text (1026):

Palladium Polymerization Procedure: (0.06 mmol scale) Polyethylene (1.3 g) was isolated as an oil. The ¹H NMR spectrum (C₆D₆) shows resonances consistent with the formation of branched polymer. Resonances consistent with olefinic end groups are observed in the baseline.

Detailed Description Text (1031):

Palladium Polymerization Procedure: (0.03 mmol scale; Isolated [(2-Me-6-chloroPh)₂DABMe]₂[PdMe(NCMe)]SbF₆ was used.) Polyethylene (3.39 g) was isolated as an oil. The ¹H NMR spectrum (C₆D₆) shows the production of branched polyethylene; internal olefin end groups are also present.

Detailed Description Paragraph Table (3):

¹³C NMR data TCB, 120.degree. C., 0.05 M CrAcAc freq ppm Intensity 46.5568
24.6005 1 cmp and/or 1, 3 ccmcc 44.9321 3.42517 1, 3 cmc 40.8118 55.4341 2 pmp
40.3658 145.916 1, 3 pmp 39.5693 18.458 methylenes from 2 cmp and/or 2 cmc 38.7782
4.16118 38.6295 5.84037 38.2844 8.43098 38.1198 8.29802 37.8384 3.83966 37.5198
13.4977 37.2384 23.4819 37.1163 16.8339 36.7446 114.983 36.0012 6.19217 35.7198
5.17495 34.2278 4.83958 32.9216 20.2781 3B.sub.6.sup.+, 3EOC 32.619 3.6086 32.4172
2.98497 32.1995 10.637 31.9765 42.2547 31.8809 143.871 30.4686 27.9974 30.3199
47.1951 30.0225 36.1409 29.7411 102.51 29.311 4.83244 28.7111 117.354 28.2597
9.05515 27.1659 22.5725 27.0067 5.81855 26.1146 13.5772 24.5642 2.59695
.beta..beta.B 22.6368 12.726 2B.sub.5.sup.+, 2EOC 20.1413 3.7815 2B.sub.3 19.7271
20.0959 1B.sub.1 17.5236 7.01554 end group 14.2528 3.03535 1B.sub.3 13.8812 12.3635
1B.sub.4.sup.+, 1EOC

Detailed Description Paragraph Table (6):

¹³C NMR data TCB, 120.degree. C., 0.05 M CrAcAc Freq ppm Intensity 50.9168
5.96663 46.3865 3.27366 1 cme and/or 1, 3 ccmcc 40.7527 40.5963 2 eme 40.567 41.9953
1, 3 eme 40.3336 45.8477 1, 3 eme 37.1985 60.1003 36.6998 41.2041 36.0579 11.2879
35.607 25.169 34.4771 19.0834 34.0845 22.8886 33.1243 20.1138 32.8962 27.6778
31.8406 75.2391 30.0263 76.2755 29.6921 170.41 28.9494 18.8754 28.647 25.8032
27.4588 22.2397 27.1086 48.0806 24.3236 3.31441 22.5783 4.64411 2B.sub.5 +, 2EOC
19.6712 43.1867 1B.sub.1 17.5546 1.41279 end group 14.3399 1.74854 1B.sub.3 13.8518
5.88699 1B.sub.4 +, 1EOC 10.9182 2.17785 2B.sub.1

Detailed Description Paragraph Table (26):

¹³C NMR Data TCB, 120.degree. C., 0.05 M CrAcAc Freq ppm Intensity 48.344
1.85262 46.5562 22.8938 1 cme and/or 1, 3 ccmcc 44.9064 10.8003 1, 3 cme 42.0842
16.824 40.7845 117.364 2 eme 40.5777 113.702 1, 3 eme 40.3336 136.742 1, 3 eme
39.5591 15.0962 methylene from 2 cmc or/and 2 cme 38.7634 18.636 38.4716 12.3847
38.2488 17.3939 37.2144 17.5837 36.721 111.057 36.2913 11.0136 35.8776 22.0367
35.6176 90.3685 34.5248 15.734 34.1959 24.7661 33.0182 14.0261 31.8671 238.301
31.4056 20.6401 30.8433 11.2412 30.4613 20.2901 30.0104 62.2997 29.7133 78.3272
29.2359 31.6111 28.9653 53.5526 28.6577 64.0528 26.9813 17.6335 26.3925 4.51208
25.9363 5.6969 24.2971 1.70709 22.9019 9.13305 2B.sub.4 22.6048 14.3641 2B.sub.5 +,
2EOC 19.7348 10.124 1B.sub.1 19.1991 2.00384 1B.sub.1 17.5811 2.28331 end group
13.8783 26.3448 1B.sub.4 +, 1EOC 12.6264 19.6468 end group 10.9501 4.96188 1B.sub.2

WEST

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L17: Entry 7 of 19

File: USPT

Jun 29, 1999

DOCUMENT-IDENTIFIER: US 5916989 A

TITLE: Polymers of C.sub.4 and higher .alpha.-olefins

Abstract Text (1):

Disclosed herein are processes for polymerizing ethylene, acyclic olefins, and/or selected cyclic olefins, and optionally selected olefinic esters or carboxylic acids, and other monomers. The polymerizations are catalyzed by selected transition metal compounds, and sometimes other co-catalysts. Since some of the polymerizations exhibit some characteristics of living polymerizations, block copolymers can be readily made. Many of the polymers produced are often novel, particularly in regard to their microstructure, which gives some of them unusual properties. Numerous novel catalysts are disclosed, as well as some novel processes for making them. The polymers made are useful as elastomers, molding resins, in adhesives, etc. Also described herein is the synthesis of linear .alpha.-olefins by the oligomerization of ethylene using as a catalyst system a combination a nickel compound having a selected .alpha.-diimine ligand and a selected Lewis or Bronsted acid, or by contacting selected .alpha.-diimine nickel complexes with ethylene.

Brief Summary Text (12):

This invention also concerns a polyethylene containing the structure (XXVII) in an amount greater than can be accounted for by end groups, and preferably at least 0.5 or more of such branches per 1000 methylene groups than can be accounted for by end groups. ##STR2##

Brief Summary Text (13):

This invention also concerns a polypropylene containing one or both of the structures (XXVIII) and (XXIX) and in the case of (XXIX) in amounts greater than can be accounted for by end groups. Preferably at least 0.5 more of (XXIX) branches per 1000 methylene groups than can be accounted for by end groups, and/or at least 0.5 more of (XXVIII) per 1000 methylene groups are present in the polypropylene. ##STR3##

Brief Summary Text (88):

This invention includes a process for the production of polyolefins, comprising contacting, at a temperature of about -100.degree. C. to about +200.degree. C., one or more monomers selected from the group consisting of ethylene, an olefin of the formula R.sup.17 CH.dbd.CH.sub.2 or R.sup.17 CH.dbd.CHR.sup.17, 4-vinylcyclohexene, cyclobutene, cyclopentene, substituted norbornene, and norbornene; with a compound of the formula ##STR9## wherein: R.sup.20 and R.sup.23 are independently hydrocarbyl or substituted hydrocarbyl;

Brief Summary Text (127):

a second compound of the formula ##STR12## and one or more monomers selected from the group consisting of ethylene, an olefin of the formula R.sup.17 CH.dbd.CH.sub.2 or R.sup.17 CH.dbd.CHR.sup.17, 4-vinylcyclohexene, cyclobutene, cyclopentene, substituted norbornene, or norbornene;

Brief Summary Text (517):

The first compound and (I) are contacted, usually in the liquid phase, and in the presence of the olefin, and/or 4-vinylcyclohexene, cyclopentene, cyclobutene, substituted norbornene, or norbornene. The liquid phase may include a compound added

just as a solvent and/or may include the monomer(s) itself. The molar ratio of first compound:nickel or palladium complex is about 5 to about 1000, preferably about 10 to about 100. The temperature at which the polymerization is carried out is about -100.degree. C. to about +200.degree. C., preferably about -20.degree. C. to about +80.degree. C. The pressure at which the polymerization is carried out is not critical, atmospheric pressure to about 275 MPa, or more, being a suitable range. The pressure may affect the microstructure of the polyolefin produced (see below).

Brief Summary Text (566):

The group T.sup.3 in (VI) was originally the group T.sup.1 in (II) or (III), or the group which included R.sup.8 in (IV). It in essence will normally be one of the end groups of the eventual polymer product. The olefinic group which is coordinated to M, R.sup.11 CH.dbd.CHR.sup.11 is normally one of the monomers, olefin, cyclic olefin, or, if Pd(II) is M, an olefinic ester or carboxylic acid. If more than one of these monomers is present in the reaction, it may be any one of them. It is preferred that T.sup.3 is alkyl and especially preferred that it is methyl, and it is also preferred that R.sup.11 is hydrogen or n-alkyl. It is also preferred that M is Pd(II).

Brief Summary Text (591):

These polymers, and others herein, can have unique structures in terms of the branching in the polymer. Branching may be determined by NMR spectroscopy (see the Examples for details), and this analysis can determine the total number of branches, and to some extent the length of the branches. Herein the amount of branching is expressed as the number of branches per 1000 of the total methylene (--CH.sub.2 --) groups in the polymer, with one exception. Methylene groups that are in an ester grouping, i.e. --CO.sub.2 R, are not counted as part of the 1000 methylenes. These methylene groups include those in the main chain and in the branches. These polymers, which are E homopolymers, have a branch content of about 80 to about 150 branches per 1000 methylene groups, preferably about 100 to about 130 branches per 1000 methylene groups. These branches do not include polymer end groups. In addition the distribution of the sizes (lengths) of the branches is unique. Of the above total branches, for every 100 that are methyl, about 30 to about 90 are ethyl, about 4 to about 20 are propyl, about 15 to about 50 butyl, about 3 to about 15 are amyl, and about 30 to about 140 are hexyl or longer, and it is preferred that for every 100 that are methyl, about 50 to about 75 are ethyl, about 5 to about 15 are propyl, about 24 to about 40 are butyl, about 5 to 10 are amyl, and about 65 to about 120 are hexyl or larger. These E homopolymers are often amorphous, although in some there may be a small amount of crystallinity.

Brief Summary Text (606):

Some of the polymers produced herein are novel because of unusual structural features. Normally, in polymers of alpha-olefins of the formula CH.sub.2=CH(CH.sub.2).sub.a H wherein a is an integer of 2 or more made by coordination polymerization, the most abundant, and often the only, branches present in such polymers have the structure --(CH.sub.2).sub.a H. Some of the polymers produced herein are novel because methyl branches comprise about 25% to about 75% of the total branches in the polymer. Such polymers are described in Examples 139, 162, 173 and 243-245. Some of the polymers produced herein are novel because in addition to having a high percentage (25-75%) of methyl branches (of the total branches present), they also contain linear branches of the structure --(CH.sub.2).sub.n H wherein n is an integer of six or greater. Such polymers are described in Examples 139, 173 and 243-245. Some of the polymers produced herein are novel because in addition to having a high percentage (25-75%) of methyl branches (of the total branches present), they also contain the structure (XXVI), preferably in amounts greater than can be accounted for by end groups, and more preferably greater than 0.5 (XXVI) groups per thousand methyl groups in the polymer greater than can be accounted for by end groups. ##STR72##

Brief Summary Text (627):

Since it is believed that many of the polymerization processes herein have characteristics that often resemble those of living polymerizations, making block polymers may be relatively easy. One method is to simply allow monomer(s) that are being polymerized to be depleted to a low level, and then adding different monomer(s) or the same combination of monomers in different ratios. This process may

be repeated to obtain polymers with many blocks.

Brief Summary Text (699):

Copolymers of linear olefins with 4-vinylcyclohexene and other dienes may generally be used for all of the applications for which the linear olefins polymers (listed above) may be used. In addition they may be sulfur cured, so they generally can be used for any use for which EPDM polymers are used, assuming the olefin/4-vinylcyclohexene polymer is elastomeric.

Detailed Description Text (368):

Polymerization of ethylene was carried out according to example 112, using the complex [(2,6-i-PrPh).sub.2 DABMe.sub.2]PdMe(OSO.sub.2 CF.sub.3) (0.068 g, 0.10 mmol) as the initiator and CHCl.sub.3 as the solvent. The reaction gave 5.98 g of low viscosity fluid (2130 equivalents of ethylene per Pd). ¹H NMR (CDCl.sub.3) 0.8-1.0 (m, CH.sub.3); 1.0-1.5 (m, CH and CH.sub.2); 1.5-1.7 (m, CH.sub.3 CH.dbd.CH--); 1.9-2.1 (broad, --CH.sub.2 CH.dbd.CHCH.sub.2 --); 5.3-5.5 (m, CH.dbd.CH--). Integration of the olefin end groups assuming one olefin per chain gives M.sub.n =630 (DP=24). A linear polymer with this molecular weight and methyl groups at both ends should have 46 methyl carbons per 1000 methylene carbons. The value measured by integration is 161, thus this polymer is highly branched.

Detailed Description Text (370):

Polymerization of ethylene was carried out according to example 112, using the complex {[(2,6-i-PrPh).sub.2 DABH]PdCH.sub.2 CH.sub.2 CH.sub.2 C(O)OCH.sub.3 }SbF.sub.6.sup.- (0.082 g, 0.10 mmol) as the initiator and CHCl.sub.3 as the solvent. The reaction gave 4.47 g of low viscosity fluid (1600 equivalents of ethylene per Pd). ¹H NMR (CDCl.sub.3) is similar to example 120. Integration of the olefin end groups assuming one olefin per chain gives M.sub.n =880 (DP=31). A linear polymer with this molecular weight and methyl groups at both ends should have 34 methyl carbons per 1000 methylene carbons. The value measured by integration is 156, thus this polymer is highly branched.

Detailed Description Text (372):

Polymerization of ethylene was carried out according to example 112, using the complex {[(2,6-i-PrPh).sub.2 DABMe.sub.2]PdCH.sub.2 CH.sub.2 CH.sub.2 C(O)OCH.sub.3 }BCl (C.sub.6 F.sub.5).sub.3.sup.- (0.116 g, 0.10 mmol) as the initiator and CHCl.sub.3 as the solvent. The reaction gave 0.278 g of low viscosity fluid, after correcting for the catalyst residue this is 0.160 g (57 equivalents of ethylene per Pd). M.sub.n estimated by integration of olefin end groups is 300.

Detailed Description Text (374):

The complex [(2,6-i-PrPh).sub.2 DABMe.sub.2]PdMeCl (0.056 g, 0.10 mmol) was loaded into a Schlenk flask in the drybox followed by 40 ml of dry toluene. A solution of ethyl aluminum dichloride (1.37 ml of 0.08M solution in o-dichlorobenzene) was added while stirring. Polymerization of ethylene was carried out using this solution according to example 112. The reaction gave 0.255 g of low viscosity fluid, after correcting for the catalyst residue this is 0.200 g (71 equivalents of ethylene per Pd). M.sub.n estimated by integration of olefin end groups is 1300.

Detailed Description Text (402):

GPC was done in THF using PMMA, standards and an RI detector except for example 133 which was done in trichlorobenzene at 135.degree. C. vs. polystyrene reference with results calculated as linear polyethylene using universal calibration theory. When pc, mer end groups could be detected by ¹H NMR (5.4 ppm, multiplet, --CH.dbd.CH--, internal double bond), M.sub.n was calculated assuming two olefinic protons per chain.

Detailed Description Text (489):

The solid complex {[(2,6-i-PrPh).sub.2 DABH.sub.2]PdMe(Et.sub.2 O)}SbF.sub.6.sup.- (0.080 g, 0.10 mmol) was added as a solid to a stirring solution of cyclopentene (1.35 g, 20 mmol) in 20 ml of dichlorobenzene in the drybox. After stirring 20 h, the slightly viscous solution was worked up by removing the solvent in vacuo to give 1.05 g sticky solid (156 equivalents of cyclopentene per Pd). ¹H NMR (CDCl.sub.3): complex spectrum from 0.6-2.6 ppm with maxima at 0.75, 1.05, 1.20, 1.55, 1.65, 1.85, 2.10, 2.25, and 2.50. There is also a multiplet for internal

olefin at 5.25-5.35. This is consistent with a trisubstituted cyclopentenyl end group with a single proton (W. M. Kelly et. al., Macromolecules 1994, 27, 4477-4485.) Integration assuming one olefinic proton per polymer chain gives DP=8.0 and M.sub.n =540. IR (Thin film between NaCl plates, cm.sup.-1): 3048 (vw, olefinic end group, CH stretch), 1646(vw, olefinic end group, R.sub.2 C=CHR trisubstituted double bond stretch), 1464(vs), 1447(vs), 1364(m), 1332(m), 1257(w), 1035(w), 946(m), 895(w), 882(w), 803(m, cyclopentenyl end group, R.sub.2 C=CHR trisubstituted double bond, CH bend), 721(vw, cyclopentenyl end group, RHC.dbd.CHR disubstituted double bond, CH bend). GPC (trichlorobenzene, 135.degree. C., polystyrene reference, results calculated as linear polyethylene using universal calibration theory): M.sub.n =1.sup.38 M.sub.w =246 M.sub.w /M.sub.n =1.79.

Detailed Description Text (640):

Toluene (30 mL), 4-vinylcyclohexene (15 mL), and 20 mg of [(2,6-i-PrPh).sub.2 DABH.sub.2]NiBr.sub.2 (0.03 mmol) were combined in a Schlenk flask under an atmosphere of ethylene. A 10% MAO solution (3 mL) in toluene was added. The resulting purple solution was stirred for 16 h. After only a few hours, polymer began to precipitate and adhere to the walls of the flask. The polymerization was quenched and the polymer precipitated from acetone. The polymer was dried in vacuo overnight resulting in 100 mg of a white solid. Characterization by proton NMR suggests incorporation of 4-vinylcyclohexene as a comonomer. .sup.1 H NMR (CDCl.sub.3) .delta. 5.64 (m, vinyl, cyclohexene), 2.0-0.9 (overlapping m, including cyclohexyl methylene, methylene (PE), methine), 0.78 (methyl, PE). There are also some minor signals in the base line that suggests incorporation of the internal olefin (cyclohexene) and free .alpha.-olefin (4-vinyl).

Detailed Description Text (726):

A 26-ma (0.033-mmol) sample of [(2,6-i-PrPh).sub.2 DAB(4-F-Ph).sub.2]NiBr.sub.2 was magnetically stirred under nitrogen in a 50-mL Schlenk flask with 25mL of dry toluene. Then 0.6 mL of polymethylalumoxane was injected, turning the orange-brown solution to a deep green-black solution. The mixture was pressurized immediately with ethylene to 152 kPa (absolute) and stirred at RT for 17 h. The reaction soon became warm to the touch; this heat evolution persisted for over an hour and the liquid volume in the Schlenk flask was observed to be slowly increasing. After 17 h, the reaction was still dark green-brown, but thicker and significantly (20%) increased in volume. The ethylene was vented; the offgas contained about 3% butenes (1-butene, 1.9%; t-2-butene, 0.6%; c-2-butene, 0.9%) by GC (30-m Quadrex GSQ Megabore column; 50-250.degree. C. at 10.degree./min). The toluene solution was stirred with 6N HCl/methanol and was separated; the toluene was rotovapped and held under high vacuum to yield 9.53 g of low-melting polyethylene wax. There seemed to be significant low-boiling species present, probably low-mw ethylene oligomers, which continued to boil off under high vacuum. .sup.1 H NMR (CDCl.sub.3 ; 60.degree. C.) of the product showed a CH.sub.2 :CH.sub.3 ratio of 206:17, which is 57CH.sub.3 's per 1000CH.sub.2 's. There were vinyl peaks at 5-5.8 ppm; if the end groups are considered to be vinyls rather than internal olefins, the degree of polymerization was about 34.

Detailed Description Text (786):

Cyclopentene was polymerized by [(2-PhPh).sub.2 DABMe.sub.2]NiBr.sub.2 and MMAO according to Example 305 to give 1.24 g polymer (728 turnovers/Ni). The polymer was pressed at 292.degree. C. to give a transparent, light gray, brittle film. DSC (25 to 320.degree. C., 10.degree. C./min, second heat): Tm(onset)=160.degree. C., Tm(end)=285.degree. C., Heat of fusion=33 J/g. .sup.1 H NMR (400 MHz, 120.degree. C., d.sub.4 -o-dichlorobenzene) is very similar to the spectrum of Example 306. Several peaks attributed to cyclopentenyl end groups were observed in the range 5.2-5.7 ppm. Integration of these peaks was used to calculate M.sub.n =2130. IR (pressed film, cm.sup.-1): 3050 (vw, olefinic end group, CH stretch), 1615(vw, olefinic end group, cis--CH.dbd.CH-- double bond stretch), 1463(vs), 1445(vs), 1362(s), 1332(s), 1306(s), 1253(m), 1128(w), 1041(w), 935(m), 895(w), 882(w), 792(w), 721(w, olefinic end group, cis--CH.dbd.CH--, CH bend). GPC (Dissolved in 1,2,4-trichlorobenzene at 150.degree. C., run at 100.degree. C. in tetrachloroethylene, polystyrene calibration): Peak MW=13,900; M.sub.n =10,300; M.sub.w =17,600; M.sub.w /M.sub.n =1.70.

Detailed Description Text (1009):

Palladium Polymerization Procedure: (0.06 mmol scale) Polyethylene (0.06 g) was isolated as an oil. ¹H NMR spectrum (C₆D₆) shows branched polyethylene along with some internal olefinic end groups.

Detailed Description Text (1016):

Nickel Polymerization Procedure: (0.02 mmol scale) Solid white polyethylene (5.9 g) was isolated. ¹H NMR spectrum (C₆D₆) shows a significant amount of branched polymer along with internal olefinic end groups.

Detailed Description Text (1017):

Palladium Polymerization Procedure: (0.06 mmol scale) Polyethylene (0.38 g) was isolated as an oil. ¹H NMR spectrum (C₆D₆) shows a significant amount of branched polymer along with internal olefinic end groups.

Detailed Description Text (1025):

Palladium Polymerization Procedure: (0.06 mmol scale) Polyethylene (1.3 g) was isolated as an oil. The ¹H NMR spectrum (C₆D₆) shows resonances consistent with the formation of branched polymer. Resonances consistent with olefinic end groups are observed in the baseline.

Detailed Description Text (1030):

Palladium Polymerization Procedure: (0.03 mmol scale; Isolated [(2-Me-6-chloroPh)₂DABMe₂]PdMe(NCMe)]SbF₆ was used.) Polyethylene (3.39 g) was isolated as an oil. The ¹H NMR spectrum (C₆D₆) shows the production of branched polyethylene; internal olefin end groups are also present.

Detailed Description Paragraph Table (3):

		¹³ C NMR data TCB, 120C, 0.05M CrAcAc									
freq ppm	intensity										
46.5568	24.6005	1	cmp								
and/or 1,3 ccmcc	44.9321	3.42517	1,3	cmc	40.8118	55.4341	2	pmp	40.3658	145.916	1,3
pmp	39.5693	18.458	methylenes from 2	cmp	and/or 2	cmc	38.7782	4.16118	38.6295		
5.84037	38.2844	8.43098	38.1198	8.29802	37.8384	3.83966	37.5198	13.4977	37.2384		
23.4819	37.1163	16.8339	36.7446	114.983	36.0012	6.19217	35.7198	5.17495	34.2278		
4.83958	32.9216	20.2781	3B.sub.6.sup.+, 3EOC	32.619	3.6086	32.4172	2.98497	32.1995			
10.637	31.9765	42.2547	31.8809	143.871	30.4686	27.9974	30.3199	47.1951	30.0225		
36.1409	29.7411	102.51	29.311	4.83244	28.7111	117.354	28.2597	9.05515	27.1659		
22.5725	27.0067	5.81855	26.1146	13.5772	24.5642	2.59695	.beta..beta.B	22.6368	12.726		
2B.sub.5.sup.+, 2EOC	20.1413	3.7815	2B.sub.3	19.7271	20.0959	1B.sub.1	17.5236				
7.01554	<u>end group</u>	14.2528	3.03535	1B.sub.3	13.8812	12.3635	1B.sub.4.sup.+, 1EOC				

Detailed Description Paragraph Table (6):

		¹³ C NMR data TCB, 120C, 0.05M CrAcAc									
Freq ppm	Intensity										
50.9168	5.96663	46.3865									
3.27366	1	cme and/or 1,3 ccmcc	40.7527	40.5963	2	eme	40.567	41.9953	1,3	eme	40.3336
45.8477	1,3	eme	37.1985	60.1003	36.6998	41.2041	36.0579	11.2879	35.607	25.169	
34.4771	19.0834	34.0845	22.8886	33.1243	20.1138	32.8962	27.6778	31.8406	75.2391		
30.0263	76.2755	29.6921	170.41	28.9494	18.8754	28.647	25.8032	27.4588	22.2397		
27.1086	48.0806	24.3236	3.31441	22.5783	4.64411	2B.sub.5 +, 2 EOC	19.6712	43.1867			
1B.sub.1	17.5546	1.41279	<u>end group</u>	14.3399	1.74854	1B.sub.3	13.8518	5.88699	1B.sub.4		
+, 1EOC	10.9182	2.17785	2B.sub.1								

Detailed Description Paragraph Table (26):

		¹³ C NMR Data TCB, 120C, 0.05M CrAcAc									
Freq ppm	Intensity										
48.344	1.85262	46.5562									
22.8938	1	cme and/or 1,3 ccmcc	44.9064	10.8003	1,3	cme	42.0842	16.824	40.7845		
117.364	2	eme	40.5777	113.702	1,3	eme	40.3336	136.742	1,3	eme	39.5591
15.0962	methylene from 2 cmc or/and 2 cme										
38.7634	18.636	38.4716	12.3847	38.2488	17.3939						
37.2144	17.5837	36.721	111.057	36.2913	11.0136	35.8776	22.0367	35.6176	90.3685		
34.5248	15.734	34.1959	24.7661	33.0182	14.0261	31.8671	238.301	31.4056	20.6401		
30.8433	11.2412	30.4613	20.2901	30.0104	62.2997	29.7133	78.3272	29.2359	31.6111		
28.9653	53.5526	28.6577	64.0528	26.9813	17.6335	26.3925	4.51208	25.9363	5.6969		
24.2971	1.70709	22.9019	9.13305	2B.sub.4	22.6048	14.3641	2B.sub.5 +, 2EOC	19.7349			
10.124	1B.sub.1	19.1991	2.00384	1B.sub.1	17.5811	2.28331	<u>end group</u>	13.8783	26.3448		

1B.sub.4 +, 1EOC 12.6264 19.6468 end group 10.9501 4.96188 1B.sub.2

CLAIMS:

6. The polymer as recited in claim 5 which contains the structure ##STR94## and wherein (XXVI) is present in an amount of 0.5 branches of (XXVI) or more per 1000 methylene atoms greater than can be accounted for by end groups.

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L19: Entry 3 of 18

File: USPT

Jul 11, 1989

DOCUMENT-IDENTIFIER: US 4847005 A

TITLE: Solutions of hydrolytically stable polymeric acetal carboxylate salts and stable liquid detergent compositions containing such salts

Detailed Description Text (6):

As further noted above, the polymer includes end cap moieties which provide stability against degradation in alkaline media. Thus, the builder polymer molecule generally corresponds to the formula: ##STR6## where R.sup.1 and R.sup.2 are end cap moieties and M and n are as defined above. By way of example, suitable chemically stable end groups include stable substituent moieties derived from otherwise stable compounds such as: alkanes such as methane, ethane, propane, butane, and higher alkanes such as decane, dodecane, cotadecane and the like; alkenes such as ethylene, propylene, butylene, decene, dodecene and the like, branch chain hydrocarbons, both saturated and unsaturated, such as 2-methyl butane, 2-methyl butene, 4-butyl-2,3-dimethyl octane and the like; aromatic hydrocarbons such as benzene, toluene, xylene, and the like; cycloalkanes, and cycloalkenes, such as cyclohexane and cyclohexene and the like; haloalkanes such as chloromethane, chlorobutane, dichloropentane and the like; alcohols such as methanol, ethanol, 2-propanol, cyclohexanol, sodium phenate, and the like; polyhydric alcohols such as 1,2-ethane diol, 1,4-benzene diol and the like; mercaptans such as methane thiol, 1,2-methanedithiol and the like, ethers such as the methoxyethane methyl ether, ethyl ether, ethoxy propane and cyclic ethers such as ethylene oxide, epichlorohydrin, tetramethylene oxide and the like; aldehydes and ketones and the like; and carboxylate-containing compounds such as the alkali metal salts of carboxylic acids, the esters of carboxylic acids and the anhydrides.

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L19: Entry 14 of 18

File: JPAB

Jun 5, 1990

DOCUMENT-IDENTIFIER: JP 02145615 A
TITLE: MODIFIED VINYLESTER RESIN

Abstract (2):

CONSTITUTION: This resin consists of (A) a terminal unsaturated vinyl ester, (B) an unsaturated polyester having cyclopentene or cyclohexene terminal group, (C) bismaleimide, (D) aminophenol as comonomer and, as occasion demands, (E) a general additive. Preferably, the component A has a group of the formula (R is H or CH

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L19: Entry 14 of 18

File: JPAB

Jun 5, 1990

PUB-NO: JP402145615A
DOCUMENT-IDENTIFIER: JP 02145615 A
TITLE: MODIFIED VINYLESTER RESIN

PUBN-DATE: June 5, 1990

INVENTOR-INFORMATION:

NAME

COUNTRY

PETER, ROLAND

HOLOCH, JAN DR

EISENBARTH, PHILIPP

ASSIGNEE-INFORMATION:

NAME

COUNTRY

BASF AG

APPL-NO: JP01265341

APPL-DATE: October 13, 1989

US-CL-CURRENT: 524/508

INT-CL (IPC): C08F 299/00

ABSTRACT:

PURPOSE: To provide a resin having satisfactory toughness and excellent heat resistance and useful as hardenable molding material by using an unsaturated polyester having cyclopentene or cyclohexane terminal group.

CONSTITUTION: This resin consists of (A) a terminal unsaturated vinyl ester, (B) an unsaturated polyester having cyclopentene or cyclohexene terminal group, (C) bismaleimide, (D) aminophenol as comonomer and, as occasion demands, (E) a general additive. Preferably, the component A has a group of the formula (R is H or CH

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